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Indian Standard

METHOD FOR DETERMINATION OF CRUDE FIBRE CONTENT IN FOOD PRODUCTS

PART II MODIFIED SCHARRER METHOD

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(*Continued from page* 1)

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Indian Standard

METHOD FOR DETERMINATION OF CRUDE FIBRE CONTENT IN FOOD PRODUCTS

PART II MODIFIED SCHARRER METHOD

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 30 July 1982, after the draft finalized by the Food Hygiene Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 This standard is based on ISO/DIS 6541-1981 'Agricultural food products — Determination of crude fibre content — Modified Scharrer method' issued by the International Organization for Standardization.

0.3 There are numerous methods for the determination of the crude fibre content of agricultural food products and for a given method, numerous variants are used, for different products to be analysed or for the same product in different laboratories. This standard has been formulated with a view to provide uniform methods of analysis and to facilitate the interpretation of results.

0.4 This standard is being issued in two parts. This part (Part II) covers the modified Scharrer method for determination of crude fibre content of agricultural food products. Part I covers, a method of general application based on the Weende method which is most commonly used for determination of crude fibre content of agricultural food products.

0.4.1 The method described in this standard is applicable to cereals and cereal products, as well as to certain products containing less than one percent of crude fibre, for example, yeasts, which are excluded from the field of application of general method (Part I).

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part II) specifies a method for the determination of the crude fibre content of agricultural food products containing less than one percent of crude fibre.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1977*) shall be used where the use of water as a reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of test.

3. TERMINOLOGY

3.1 For the purpose of this standard, the crude fibre shall be the material which is insoluble and combustible under the conditions of test.

3.2 The crude fibre content is expressed as a percentage by mass, calculated either on the basis of the product as received or on the basis of the dry matter content of the product.

4. PRINCIPLE

4.1 After grinding and defatting, if necessary, the product is boiled with a mixture of acetic acid, nitric acid and trichloroacetic acid (Scharrer reagent). The insoluble residue is separated and washed on a filter crucible. The insoluble residue is dried and weighed. Loss of mass on incineration is then determined.

5. APPARATUS

5.1 Grinding Device — Easy to clean, suited to the nature of product and allowing grinding of the product without causing undue heating and without loss on absorption of moisture.

5.2 Sieve — It shall be of metal wire cloth, of one mm nominal aperture size, complying with the requirements of IS : 460 (Part I)-1978†.

5.3 Wide Mouthed Vessel — Provided with a condenser, for example, a conical flask of capacity 200 to 300 ml fitted with a reflux condenser.

5.4 Suction Flask

*Specification for water for general laboratory use (*second revision*).

†Specification for test sieves: Part I Wire cloth test sieves (*second revision*).

5.5 Water-Jet Pump

5.6 Filter Crucible — Silica crucible, having a fitted silica plate of mean pore diameter 40 to 90 μm prepared as given in **5.6.1** and **5.6.2.**

5.6.1 Prior to use, carefully clean the filter crucible and heat in the muffle furnace controlled at 550 ± 25°C, for 6 hours.

5.6.2 Spread on the crucible plate, 5 to 6 g of sea sand (*see* **6.3**) weighed to the nearest one mg. Level the surface and spread over the top, 4 to 5 g of crushed porcelain and again level the surface. Using gentle pressure, place a perforated porcelain (*see* **6.4**) disc on top of these two layers. This filling may be reused without cleaning of preliminary treatment, but care shall be taken to ensure that the layers remain in their initial positions.

5.7 Desiccator — It shall be of diameter less than 25 cm containing an efficient desiccant.

5.8 Drying Oven — Capable of being controlled at 130 to 133°C.

5.9 Muffle Furnace — Capable of being controlled at 550 ± 25°C.

5.10 Analytical Balance

6. REAGENTS

6.1 Scharrer Reagent — A mixture consisting of 900 ml aqueous solution of acetic acid (density 1·07 g/ml at 27°C), 60 ml of nitric acid (density 1·4 g/ml at 27°C) and 24 g of crystalline trichloroacetic acid.

NOTE — The acetic acid solution is prepared by diluting 730 g of 96 percent (*m/m*) glacial acetic acid with water to 1 000 g.

6.2 Acetone

6.3 Sea Sand — It shall be of particle size less than 0·5 mm, prepared as given in **6.3.1.**

6.3.1 Heat the sand with 4 N hydrochloric acid solution to boiling to eliminate iron; wash with water till the washings are free from chlorides, as indicated by the lack of a reaction with silver nitrate solution; and incinerate in the muffle furnace (*see* **5.9**) at 550 ± 25°C for 6 to 9 hours.

6.4 Boiling Aids — For example, crushed porcelain.

7. SAMPLING

7.1 Prepare a representative and homogeneous sample as appropriate for the specific product to be analysed.

8. PROCEDURE

8.1 Preparation of Test Sample

8.1.1 *Preliminary Drying* — In case of products which cannot be mixed ground as such due to high moisture content, carry out a preliminary drying of the product at an appropriate temperature. In this case, weigh the product before the preliminary drying and again just before preparation of the test sample.

8.1.2 *Products Not Requiring Grinding* — Products of which 95 percent pass through the sieve (**5.2**) need not be ground before analysis. Mix well before taking the test portion.

8.1.2.1 If the results are to be expressed relative to the dry matter, determine before hand, the dry matter content of the test sample (*see* **8.1.2**) by the appropriate method.

8.1.3 *Products Requiring Grinding* — Products of which less than 95 percent pass through the sieve (**5.2**) shall be ground.

8.1.3.1 If the results are to be expressed relative to the product as received, determine beforehand the dry matter content of the sample by the appropriate method.

8.1.3.2 Grind the laboratory sample in the grinding device (**5.1**) so that at least 95 percent of the product passes through the sieve (**5.2**).

NOTE — Such a degree of fineness of grinding is unnecessary for products containing parts which are rich in cellulosic substances, such as grains in husk or husks of grain.

8.1.3.3 Determine the dry matter content of the test sample (*see* **8.1.3.2**) by the appropriate method.

8.2 Test Portion — Weigh, to the nearest one mg, a mass of the prepared test sample (**8.1**) corresponding to 0·05 to 0·15 g of crude fibre.

8.3 Determination — Products rich in fatty substances may require defatting beforehand in accordance with the methods described in Annexure A of IS : 10226 (Part I)-1982/ISO 5498-1981*. This operation is not required for cereal, cereal products and yeasts.

8.3.1 *Digestion*

8.3.1.1 Transfer the test portion to the digestion vessel (**5.3**) and suspend it in about one-third of the total volume of the Scharrer reagent (**6.1**). Generally, the total valume, in millilitres, of the Scharrer reagent

*Agricultural food products — Determination of crude fibre content — General method.

(**6.1**) used is numerically twenty times the mass, in grams, of the test portion, however, in no case shall the volume used be less than 40 ml.

8.3.1.2 Using a glass rod, which shall be left in the vessel (**5.3**), break up any large lumps that may have been formed.

8.3.1.3 Carefully rinse the interior walls of the vessel (**5.3**) with the remaining two thirds of the Scharrer reagent in order to remove any particles of the product adhering to the walls. Fit the condenser. Bring the contents of the vessel to the boil in 3 $\pm$ 0·5 minutes and maintain boiling for 30 $\pm$ 1 minutes (*see* Note). Do not stir or shake while boiling.

NOTE — During digestion heating shall be none with care in order to avoid overheating and too rapid boiling. The foam formed in the vessel shall never be allowed to exceed a height of 10 mm. Control may be exerted by varying the intensity of heating.

8.3.2 *Separation and Washing of the Residue*

8.3.2.1 After the specified boiling period, transfer the boiling solution to the filter crucible (**5.6**) prepared as described in **5.6** and **5.6.2** and filter under reduced pressure using the suction flask (**5.4**) and the water-jet pump (**5.5**).

8.3.2.2 Rinse the vessel and the glass rod with 50 to 70 ml portions of water (temperature between 95 and 100°C) and quantitatively transfer the insoluble residue to the filter crucible using a glass rod fitted with a rubber cap. Repeat the washing until the filterate is substantially neutral to litmus paper; this usually requires 300 to 400 ml water.

8.3.2.3 After washing, disconnect the water jet pump, immediately empty the suction flask and fill the filter crucible three times with the acetone (**6.2**), allowing the solvent to drain through, under gravity. If this operation requires too much time, apply gentle suction to obtain a flow rate not exceeding one drop per second.

8.3.3 *Drying*

8.3.3.1 Dry the filter crucible with its contents in the drying oven (**5.8**) maintained at 130 to 133°C (*see* Note).

8.3.3.2 Allow to cool to room temperature in the desiccator (**5.7**) and weigh rapidly to the nearest 0·5 mg.

8.3 3.3 Repeat these operations until the difference between two successive weighings, separated by a period in the oven followed by cooling in the desiccator, does not exceed one mg.

NOTE — Drying for one hour is generally sufficient.

8.3.4 *Incineration*

8.3.4.1 Place the crucible and its contents in the muffle furnace (**5.9**) maintained at 550 ± 25°C and incinerate the dry residue at this temperature for 30 min.

8.3.4.2 Place the crucible on a refractory plate and allow to cool to room temperature in the desiccator (**5.7**). Then weigh rapidly to the nearest 0·5 mg.

8.3.5 *Number of Determinations* — Carry out at least two determinations on the same test sample.

9. EXPRESSION OF TEST RESULTS

9.1 Method of Calculation and Formulae

9.1.1 The crude fibre content, expressed as a percentage by mass relative to the product as received, is calculated as under :

9.1.1.1 Products not requiring grinding:

$$\text{Crude fibre content, percent by mass} = (m_1 - m_2) \times \frac{100}{m_0}$$

9.1.1.2 Products requiring grinding:

$$\text{Crude fibre content, percent by mass} = (m_1 - m_2) \times \frac{100}{m_0} \times \frac{100}{M_s'} \times \frac{M_s}{100}$$

9.1.2 The crude fibre content, expressed as a percentage by mass relative to the dry matter content of the product, is calculated as follows:

$$\text{Crude fibre content, percent by mass} = (m_1 - m_2) \times \frac{100}{m_0} \times \frac{100}{M_s}$$

where

m_0 = mass in grams of the test portion (*see* **8.2**);

m_1 = total mass in grams, of the dry residue and its support after drying (*see* **8.3.3**) and before incineration;

m_2 = total mass in grams, of the dry residue and its support after incineration (*see* **8.3.4**);

M_s = dry matter content of the test sample as received, determined as indicated in **8.1.3.1**; and

M_s' = dry matter content of the test sample, determined as indicated in **8.1.2.1** or **8.1.3.3.**

9.1.3 If preliminary drying was carried out (**8.1.1**), the crude fibre content, expressed as a percentage by mass relative to the product as received, is obtained by multiplying the result calculated according to **9.1.1** by the ratio:

$$\frac{m_5}{m_4}$$

where

m_4 = mass in grams, of the initial moist sample before preliminary drying, and

m_5 = mass in grams, of the sample after preliminary drying.

9.1.4 Take as the result, the arithmetic mean of two determinations, provided that the requirements for repeatability (*see* **9.2**) are satisfied.

9. Repeatability — The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst, shall not exceed:

a) 0·1 (absolute value) for crude fibre contents below 2 percent (*m/m*).

b) 5 percent (relative value) for crude fibre contents greater than 2 percent (*m/m*).

INDIAN STANDARDS

ON

FOOD ANALYSIS

IS:

5398-1969 Methods for estimation of thiamine (vitamin B_1) in food stuffs

5399-1969 Methods for estimation of riboflavin (vitamin B_2) in foodstuffs

5400-1969 Methods for estimation of nicotinic acid (niacin)

5401-1969 Methods for detection and estimation of coliform bacteria in foodstuffs

5402-1969 Method for plate count of bacteria in foodstuffs

5403-1969 Method for yeast and mould count in foodstuffs

5835-1970 Method for estimation of vitamin D in foodstuffs

5838-1970 Method for estimation of vitamin C in foodstuffs

5886-1970 Methods for estimation of carotenes and vitamin A (retinol) in foodstuffs

5887 Methods for detection of bacteria responsible for food poisoning:

(Part I)-1976 Isolation, identification and enumeration of *Escherichia coli*

(Part II)-1976 Isolation, identification and enumeration of *Staphylococcus-aureus* and *faecal Streptococci*

(Part III)-1976 Isolation and identification of *Salmonella* and *Shigella*

(Part IV)-1976 Isolation and identification of *Clostridium welchii, Clastridium Botulinum* and *Bacillus cereus* and enumeration of *Clostridium welchii* and *Bacillus cereus*

(Part V)-1976 Isolation, identification and enumeration of *Vibrio cholerae* and *Vibrio Parahae-molyticus*

7219-1973 Method for determination of protein in foods and feeds

7234-1974 Method for estimation of folic acid in foodstuffs

7235-1974 Method for estimation of tocopherol (vitamin E) in foodstuffs

7529-1975 Method for estimation of vitamin B_{12} in foodstuffs

7530-1975 Method for estimation of pyridoxine (vitamin B_6) in foodstuffs

7815-1975 Method for estimation of amino acids in food

8168-1976 Method for determination of available lysine in foods

10226 (Part I)-1982 Method for determination of crude fibre content: Part I General Method